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TAKAYANAGI YASUYUKI**(54) PRODUCTION OF IMINODISUCCINIC ACID AND ITS ALKALI METAL SALT AND A BIODEGRADABLE CHELATING AGENT CONTAINING THE SAME**

(57)Abstract:

PURPOSE: To obtain high-purity (free from D,D-form) iminodisuccinic acid and an alkali metal salt thereof in high yield and in a short reaction time compared to the conventional processes therefor.

CONSTITUTION: A tetraalkali metal salt of iminodisuccinic acid is obtained by adding a half ester of maleic acid to aspartic acid or ammonia under an alkaline condition followed by hydrolysis and evaporation to dryness. The second objective iminosuccinic acid is obtained by the above addition reaction followed by hydrolysis and then addition of sulfuric acid (without conducting an evaporation to dryness). In the above processes, use of L-aspartic acid in place of the ordinary aspartic acid produces D,D-form-free iminodisuccinic acid and tetraalkali metal salt thereof. Or, L,L-iminodisuccinic acid is selectively obtained by prior crystallization and/or washing of a mixture of the L,L-form and D,D-form of iminodisuccinic acid or a tetraalkali metal salt thereof. And, the other objective biodegradable chelating agent contains, as active ingredient, the D,D-form-free

iminodisuccinic acid and/or an alkali metal salt thereof.

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CLAIMS

[Claim(s)]

[Claim 1] The manufacture approach of the alkali-metal salt of the imino disuccinic acid characterized by adding maleic-acid half ester to an aspartic acid or ammonia under alkaline conditions.

[Claim 2] The manufacture approach of the imino disuccinic acid characterized by adding maleic-acid half ester to an aspartic acid or ammonia under alkaline conditions, and subsequently carrying out **** crystallization by the mineral acid.

[Claim 3] (L) The manufacture approach of the alkali-metal salt of the imino disuccinic acid which does not include - object characterized by adding maleic-acid half ester to - aspartic acid under alkaline conditions (D, D).

[Claim 4] (L) The manufacture approach of the imino disuccinic acid which does not include - object characterized by adding maleic-acid half ester to - aspartic acid under alkaline conditions, and subsequently carrying out **** crystallization by the mineral acid (D, D).

[Claim 5] The alternative acquisition approach of - imino disuccinic acid characterized by clearance of - object by precedence crystallization (D, L), or elution of - object by backwashing by water (L, L) out of the mixture of (L, L)-object of imino disuccinic acid or its alkali-metal salt, and (D, L) - object (L, L), or its alkali-metal salt.

[Claim 6] The imino disuccinic acid which does not include (D, D)-object, and the biodegradability chelating agent of the alkali-metal salt which contains a kind as an active principle at least.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the approach of manufacturing imino disuccinic acid and its alkali-metal salt, from maleic-acid half ester, ammonia or maleic-acid half ester, and an aspartic acid.

[0002] Moreover, this invention relates to the approach of manufacturing (L)-aspartic acid, the imino disuccinic acid which does not include (D, D)-object from maleic-acid half ester, and its alkali-metal salt.

[0003] This invention relates to the utilization as a biodegradability chelating agent of the imino disuccinic acid which does not include (D, D)-object, and its alkali-metal salt further again. Imino disuccinic acid and its alkali-metal salt are widely used for a cleaning agent constituent, a detergent builder, a heavy-metal blockade agent, a peroxide stabilizer, etc. as a biodegradability chelating agent.

[0004]

[Description of the Prior Art] The method of manufacturing imino disuccinic acid is variously learned from the former. For example, there are an approach (British patent specification No. 1306331) of adding the maleic acid of dyad to an ammonia monad, the approach (JP,5-320109,A) of adding the maleic acid of a monad to an aspartic-acid monad, etc.

[0005] The greatest trouble of these conventional technique is in refractory [of a maleic acid]. In manufacture of imino disuccinic acid, in order to obtain practical reaction yield, it is necessary to carry out the long duration reflux of the reaction mixture. However, on such manufacture conditions, it is accompanied by side reaction, such as decomposition to isomerization to the fumaric acid of a maleic acid, the hydration to the malic acid of a maleic acid, or the aspartic acid of imino disuccinic acid. It not only reduces the yield of the specified substance, but isomerization and hydration of a maleic acid become the cause of often giving coloring to reaction mixture. Moreover, decomposition of imino disuccinic acid is based on a reverse Michael addition reaction, and often causes racemization of the specified substance.

[0006] Another trouble of the conventional technique is that the imino disuccinic acid made into the object is obtained as mixture of a stereoisomer. That is, being obtained as stereoisomer mixture of 75% of (D, L)-objects, 25% of (D, D)-objects, and 25% of (L, L)-objects has obvious imino disuccinic acid obtained by the conventional technique which adds the maleic acid of dyad to an ammonia monad from the reaction mechanism. Moreover, similarly, unless the conventional technique which adds the maleic acid of a monad to an aspartic-acid monad also uses (L)-aspartic acid for a raw material, the stereoisomer mixture of the imino disuccinic acid obtained contains very scarce (D, D) - object 25% in biodegradability.

[0007] Further, (D, L)-imino disuccinic acid, and (D, D)- or (L, L) its - imino disuccinic acid is that reference is not made at all by these chelate force or water-soluble differences, although another trouble has the relation of a different diastereomer of a property of the conventional technique chemically. Namely, as for possibility of separating and using biodegradability, water solubility, the chelate force, -

(L, L) imino disuccinic acid that was excellent also from especially the standpoint of *****, and its alkali-metal salt, examination is not yet tried.

[0008]

[Problem(s) to be Solved by the Invention] This invention was made that the trouble of these conventional technique should be solved, aims at offering the advantageous manufacture approach industrially [imino disuccinic acid and its alkali-metal salt], and, specifically, makes it a technical problem to offer the imino disuccinic acid made into the object, and its alkali-metal salt by short reaction time and high yield, and the high grade by using ammonia or an aspartic acid as a raw material, and using maleic-acid half ester excellent in reactivity for an addition reaction.

[0009] Moreover, excel in biodegradability (L, L), or (D, L) this invention aims at offering the advantageous alternative manufacture approach industrially [- imino disuccinic acid and its alkali-metal salt]. By using (L)-aspartic acid as a raw material and specifically using maleic-acid half ester excellent in reactivity for an addition reaction Or you consider as the object (D, L) (L, L), let it be a technical problem not to be accompanied by the byproduction of (D, D)-object but to offer - imino disuccinic acid and its alkali-metal salt by short reaction time and high yield, and the high grade.

[0010] Furthermore, let it be a technical problem to offer the advantageous separation approach industrially [biodegradability, the chelate force, - imino disuccinic acid that is excellent in especially water solubility (L, L), and its alkali-metal salt] from the stereoisomer mixture of this invention and (L, L) (D, L) - imino disuccinic acid.

[0011]

[Means for Solving the Problem] this invention persons are replacing with the maleic acid used conventionally and using maleic-acid half ester, in case an addition reaction's is wholeheartedly presented with an aspartic acid or ammonia under alkaline conditions as a result of research said technical problem's being solved, and the alkali-metal salt of the imino disuccinic acid made into the object found out being obtained with high reaction yield in spite of short reaction time. Similarly, when presenting an addition reaction with (L)-aspartic acid under alkaline conditions, by using maleic-acid half ester, in spite of short reaction time, it considers as the object (L, L), and (D, L) the 1:1 stereoisomer mixture of - imino disuccinic acid alkali-metal salt found out being obtained with high reaction yield. Moreover, when (L)-aspartic acid was used as a raw material in addition to the content of by-products, such as a fumaric acid in these resultants, a malic acid, and a maleic acid, being low concentration very much, since (D, D)-imino disuccinic acid was not generating at all, it found out that the 4 alkali-metal salt of only only carrying out evaporation to dryness of the resultant and (L, L) (D, L) - imino disuccinic acid was obtained by high yield and the high grade.

[0012] Furthermore, while finding out that the crystal of imino disuccinic acid is obtained by adding a mineral acid after concentration suitably in these resultants, this invention persons When (L)-aspartic acid was especially used as a raw material, it found out after **** that (L, L)-imino disuccinic acid or its alkali-metal salt was selectively acquirable according to elution of - object by clearance of - object by precedence crystallization (D, L), or washing using water (L, L) etc. And it found out that it was the chelating agent is obtained (L, L) and (D, L) - imino disuccinic acid and its alkali-metal salt excel [chelating agent] in biodegradability extremely by these approaches. Moreover, (L, L)-imino disuccinic acid or its alkali-metal salt finds out that it is the chelating agent which is excellent from especially any standpoint of biodegradability, water solubility, and the chelate force etc., and reaches this invention.

[0013] That is, this invention adds the manufacture approach of the alkali-metal salt of the imino disuccinic acid characterized by adding maleic-acid half ester to an aspartic acid or ammonia under alkaline conditions, and maleic-acid half ester to ammonia or an aspartic acid under alkaline conditions, and relates to the manufacture approach of the imino disuccinic acid characterized by subsequently carrying out **** crystallization by the mineral acid.

[0014] Similarly, this invention adds the bottom maleic-acid half ester of alkaline conditions to the manufacture approach of the alkali-metal salt of the imino disuccinic acid which does not include - object characterized by adding the bottom maleic-acid half ester of alkaline conditions to (L)-aspartic acid (D, D), and (L)-aspartic acid, and relates to the manufacture approach of the imino disuccinic acid

which does not include - object characterized by subsequently carrying out **** crystallization by the mineral acid (D, D).

[0015] Furthermore, this invention out of the mixture of (L, L)-object of imino disuccinic acid or its alkali-metal salt, and (D, L) - object the acquisition approach of - imino disuccinic acid characterized by alternative elution of - object by alternative clearance of - object by precedence crystallization (D, L), or washing (L, L) (L, L), or its alkali-metal salt -- and It is related with the biodegradability chelate which contains at least one kind of the imino disuccinic acid which does not include (D, D)-object, and its alkali-metal salt as an active principle.

[0016] This invention is explained below at a detail. The approach of this invention consists of an addition reaction process which makes maleic-acid half ester act on an aspartic acid or ammonia under alkaline conditions, and the hydrolysis reaction process which adds an alkali-metal hydroxide to the resultant and the evaporation-to-dryness process which isolates the imino disuccinic acid 4 alkali-metal salt which condenses the resultant thoroughly further and is made into the object. Or it consists of an addition reaction process which makes maleic-acid half ester act on an aspartic acid or ammonia under alkaline conditions, and the hydrolysis reaction process which adds an alkali-metal hydroxide to the resultant and the **** crystallization process of adding a mineral acid to the resultant further, and isolating imino disuccinic acid.

[0017] Moreover, when (L)-aspartic acid is used for an addition reaction process, the approach of this invention consists of a precedence crystallization clearance process which carries out the crystal of (D, L)-object a ** exception from the stereoisomer mixture of - obtained according to a **** process (L, L), and (D, L) - imino disuccinic acid, and acquires - (L, L) imino disuccinic acid selectively. Or it consists of a washing process which washes with water the stereoisomer mixture of - obtained according to an evaporation-to-dryness process (L, L), and (D, L) a - imino disuccinic acid 4 alkali-metal salt, and acquires selectively a - (L, L) imino disuccinic acid 4 alkali-metal salt.

[0018] Although the maleic-acid half ester used as a raw material of this invention can be chosen from half-ester with the low-molecular alcohol of about 1-5 carbon numbers, half-ester with methyl alcohol has methyl, ethyl, propyl, isopropyl, and especially preferably desirable half-ester with the alcohol of butyl from the standpoint of the ease of preparation, reactivity, water solubility, and cost.

[0019] Maleic-acid half ester may be prepared as follows in advance of the reaction of this invention, although what was compounded by what kind of process is usable. That is, maleic-acid half ester is preferably compounded near the boiling point of low-molecular alcohol one to 5 times to a maleic anhydride for 0.1 to 3.2 hours a mol and by carrying out heating reflux for 0.2 to 0.7 hours under coexistence with the low-molecular alcohol of a 1.0 to 1.5 time mol. Usually, maleic-acid half ester is quantitatively obtained to a maleic anhydride as a transparent and colorless liquid by distilling off superfluous low-molecular alcohol under ordinary pressure or reduced pressure conditions.

[0020] The obtained maleic-acid half ester may once be isolated, or may be used for degree process as it is within a reaction vessel. Although superfluous low-molecular alcohol does not necessarily need to be distilled off, if there is much ullage, adverse effects, such as lowering of the reaction temperature in degree process, may be done. Maleic-acid half ester can be diluted and used using an undiluted solution or its outstanding water solubility. Or a twice [equimolar] as many alkali-metal hydroxide as this neutralizes, and an effective means also uses as a maleic-acid half ester 1 alkali-metal salt water solution.

[0021] Although D object, L bodies, and racemic modification can be used for the ARUPARAGIN acid used in an addition reaction process, L bodies are preferably chosen from a viewpoint of the biodegradability of a product. When it is going to obtain the imino disuccinic acid which does not include disadvantageous (D, D) - object on biodegradability, it is desirable to choose (L)-aspartic acid as a raw material and to present an addition reaction with maleic-acid half ester. 70% or more of purity which can come to hand industrially, although 85% or more of solid-state can be preferably used for the ARUPARAGIN acid used in an addition reaction process, the alkali-metal salt which it may be in the middle of the manufacture, or the water solution of the alkali-metal salt can also be directly used for it.

[0022] the amount of the ARUPARAGIN acid used used in an addition reaction process -- maleic-acid

half ester -- receiving -- a 0.6 to 1.4 time mol -- desirable -- a 0.8 to 1.2 time mol -- it is especially chosen in the range of a mol 0.9 to 1.1 times preferably.

[0023] the case where ammonia is used for a raw material in an addition reaction process -- a gas, a liquid, a water solution, etc. -- various -- an activity, with a gestalt -- possible -- the amount used -- maleic-acid half ester -- receiving -- a 0.3 to 0.8 time mol -- it is preferably chosen suitably in the range of a mol 0.4 to 0.6 times.

[0024] The alkali conditions in an addition reaction process are attained by addition of an alkali-metal hydroxide or its water solution. In addition, an aspartic-acid alkali-metal salt, maleic-acid half ester alkali-metal salts, and these water solutions may be used. as the class of alkali metal to be used -- Li, Na, or K -- K and Na are used preferably. pH of the alkaline conditions in an addition reaction process -- 10-13 -- it is preferably chosen in 10.5-12.5. By 13 or more pH, since the alkali-metal hydroxide of the molar quantity exceeding the sum total of the carboxyl group in reaction mixture exists, the hydrolysis to a maleic-acid 2 alkali-metal salt from a maleic-acid half ester 1 alkali-metal salt advances. Since it will lead to lowering of the addition reaction rate as the whole if the byproduction rate of a maleic-acid 2 alkali-metal salt increases, too much alkaline conditions must be avoided.

[0025] Three persons of the alkali-metal salt for attaining an aspartic acid or ammonia, maleic-acid half ester, and alkaline conditions in advance of an addition reaction process are mixed underwater. One of the points the approach of this invention using maleic-acid half ester is excellent in compared with the conventional technique is that a combination broad to the mixed sequence foreword and approach is possible. That is, although the process which hydrates a maleic-acid anhydride in a maleic acid in a water solution beforehand was required of the conventional technique, before this hydration was fully performed, when an aspartic acid or ammonia was mixed, there was a fault that the amidation to the amino group will advance preferentially. On the other hand, according to the approach of **** this invention for maleic-acid half ester, there is no fear of amidation of side reaction advancing under any conditions. Maleic-acid half ester is in the point which is the liquid of the non-volatile which is rich in water solubility and a fluidity to a still more advantageous point being a solid-state with a maleic acid and a maleic-acid anhydride inconvenient to handling.

[0026] The temperature in an addition reaction process is preferably good to carry [80-150-degree C] out in 100-120 degrees C. Although a reaction is usually carried out by ordinary pressure, it is a means also with effective also carrying out under application-of-pressure-among autoclave conditions. Especially the application-of-pressure reaction is advantageous when using ammonia for a raw material.

[0027] The reaction time in an addition reaction process is preferably good to carry out in 2 - 5 hours still more preferably for 1 to 8 hours for 0.5 to 24 hours. One of the points most improved in this invention approach as compared with the conventional technique is that an addition reaction ends in a surprising short time. The cause by which the reactivity of a maleic-acid half ester 1 alkali-metal salt increases as compared with a maleic-acid 2 alkali-metal salt is guessed for being easier to receive addition of the amino group as a nucleophile in order for the electron density on conjugated double bond to fall. On the other hand, although the addition reaction was similarly considered about a diester object like maleic-acid dimethyl, the diester object was lacking in water solubility, and the reaction did not advance at all. That is, the maleic-acid half ester used in this invention is concluded in case of the raw material excellent in the reactivity which doubles and has high electrophilicity and high water solubility.

[0028] The maleic-acid half ester and the low-molecular alcohol of an equimolecular amount which were used at the addition reaction process 0.9 to 1.3 times to the maleic-acid half ester used at the addition reaction process a mol and by adding the metal hydroxide of a mol or its water solution in reaction mixture 1.0 to 1.05 times preferably separate, and the 4 alkali-metal salt of imino disuccinic acid generates a hydrolysis process in reaction mixture.

[0029] 80-150 degrees C of temperature in a hydrolysis process are preferably carried out in 100-120 degrees C, and reaction time is preferably good to carry out in 0.5 - 1.5 hours for 0.1 to 3.0 hours. Since it leads to the decomposition and racemization of the specified substance by reverse Michael addition, carrying out long duration processing of the reaction mixture at the elevated temperature beyond the

need should avoid.

[0030] In the approach of this invention, the reaction mixture after a hydrolysis process is always transparent and colorless, and, moreover, most impurities are not usually detected. This is very contrastive with the data which rough reaction mixture often wears coloring in the conventional technique, and contain impurities, such as a fumaric acid, a maleic acid, a malic acid, and an aspartic acid.

[0031] Furthermore, the 4 alkali-metal salt of the imino disuccinic acid obtained when an important point results in a hydrolysis process in an addition reaction process in the approach of this invention, using (L)-aspartic acid and maleic-acid half ester as a raw material is 1:1 mixture of (D, L)-object and (L, L)-object, and is that (D, D)-object is not detected at all. This is very contrastive with the data in which (D, D)-object carries out a byproduction by racemization, even when (L)-aspartic acid and a maleic acid are used as a raw material in the conventional technique. That is, manufacture of the 4 alkali-metal salt of the imino disuccinic acid which does not include (D, D)-object is realizable with the association of the (L)-aspartic acid and maleic-acid half ester in this invention approach.

[0032] The manufacture approach of the 4 alkali-metal salt of the imino disuccinic acid which does not include - object in the approach of this invention (D, D) is what has a very large meaning from the standpoint which supplies the chelating agent excellent in biodegradability. - object which has the spacial configuration of a non-natural mold in two places of a molecule (D, D) is because it is very lacking in biodegradability as compared with (L, L)-object or - (D, L) object, so it accumulates into an environment. That is, that - (D, D) object mixes in a production process has serious effect on lowering of the biodegradability of a chelating agent.

[0033] In any case, although it is attained by presenting spray dry after it carries out evaporation to dryness of the evaporation-to-dryness process or it half-condenses reaction mixture as it is by distilling off the moisture of the reaction mixture obtained according to the hydrolysis process, it is acquired by the high grade and high yield as a crystal with the snow-white 4 alkali-metal salt of imino disuccinic acid. Moreover, isomerization or decomposition of the specified substance in an evaporation-to-dryness process are not accepted.

[0034] A washing process washes the 4 alkali-metal salt of the imino disuccinic acid obtained according to the evaporation-to-dryness process using the water of optimum dose on suction filtration equipment, or is attained by making the water of optimum dose carry out the partial dissolution, and using the 4 alkali-metal salt of imino disuccinic acid as it a supernatant, residue, and ** exception. 200 - 500 % of the weight and 0-100 degrees C of water temperature are preferably carried out for the amount of the water used in a washing process suitably in 10-70 degrees C 50 to 950% of the weight to the 4 alkali-metal salt of imino disuccinic acid.

[0035] In this invention method, it is that (L, L)-object is far preferential in the filtrate from which the generation ratios of - object and (L, L)-object whose surprising things are originally 1:1 (D, L) are collected at a washing process, and this data means that the water solubility of (L, L)-object is far excellent as compared with (D, L)-object. By condensing the filtrate obtained at a washing process, it is possible to acquire selectively the 4 alkali-metal salt of (L, L)-imino disuccinic acid.

[0036] Even when separation of (D, L)-object is not performed thoroughly, the presentation ratio of (L, L)-object however, the water solution of the isomer mixture which is 60% or more preferably 55% or more in order that it may be highly transparent and after the neglect for several months may not show even the indication of slurring or discoloration clearly in comparison with this concentration water solution of the 1:1 isomer mixture by the conventional technique -- a builder -- service water -- in case it uses for a solution etc., it is very advantageous to handling.

[0037] according to the test result using the sample selectively acquired by the approach of this invention -- the 4 alkali-metal salt of (L, L)-imino disuccinic acid -- biodegradability, water solubility, and the chelate force -- it is the chelating agent which is excellent from especially any standpoint. It is suggested that it originates in two succinic-acid units projecting in the same direction to a molecule flat surface to two succinic-acid units projecting forward and backward to a molecule flat surface, respectively in the case of (D, L)-object, as for this in the case of (L, L)-object. That is, it is considered

by the molecular structure target to a water molecule or a cation to be reasonable and for (L, L)-object to have possible 5 seat coordination.

[0038] the case where (**) -aspartic acid and ammonia are used in an addition reaction process -- a washing process -- setting - (D, L) object -- it is as possible as or (L, L) to acquire selectively each of the racemic mixture which consists of a (D, D)-object.

[0039] As a mineral acid used in a **** crystallization process, although a hydrochloric acid, a nitric acid, a sulfuric acid, etc. are mentioned, especially a sulfuric acid is used preferably. A sulfuric acid is industrially chosen from the thing of 60 - 98% of available purity, and pH 1.0-3.0 and the initial complement for adjusting to pH 1.5-2.5 preferably are used in the reaction mixture obtained according to the hydrolysis process. The temperature at the time of sulfuric-acid dropping is preferably good to carry [10-100-degree C] out in 40-80 degrees C, and the range of drop time of 1 - 5 hours is preferably good for 0.5 to 6 hours.

[0040] The imino disuccinic acid which is the specified substance is obtained by collecting the crystals which deposit by suction filtration, after 0-50 degrees C ripens the reactant after sulfuric-acid addition preferably at 10-40 degrees C for 1 to 5 hours for 0 to 72 hours. The crystal obtained consists of mixture of (D, L)-object of imino disuccinic acid, and (L, L)-object, when (L)-aspartic acid is used at an addition reaction process. A by-product hardly exists.

[0041] In the approach of this invention, a surprising thing is that (D, L)-object is far preferential as the crystal from which it is obtained according to a **** crystallization process that the generation ratio of an after [a hydrolysis process (D, L)]-object and (L, L)-object is 1:1. It means eluting in filtrate - object which is excellent in water solubility (L, L) to - objects which excel [this] in crystallinity (D, L) being collected preferentially.

[0042] It not only acquires (D, L)-object and (L, L)-object of imino disuccinic acid which are made into the object as a crystal of equivalent mixture, but at a **** crystallization process, it can raise the ratio of (D, L)-object under crystal, and (L, L)-object in filtrate from the reaction mixture after **** using the principle of precedence crystallization. Thus, it is possible by carrying out a **** crystallization process as a precedence crystallization clearance process to remove a sodium sulfate and (D, L)-object and to acquire (L, L)-imino disuccinic acid selectively.

[0043] The concentration of the imino disuccinic acid in the acid coacervation in a precedence crystallization clearance process is preferably chosen from 20 - 65% of range five to 85% of the weight by the acid conversion concentration of stereoisomer mixture. The maturing temperature and maturing time in a precedence crystallization clearance process are carried out on the same conditions as a **** crystallization process.

[0044] And judgment of (L, L)-object of imino disuccinic acid and (D, L) - object was once isolated according to the evaporation-to-dryness process besides a precedence crystallization clearance process (D, L) (L, L), the stereoisomer mixture of the 4 alkali-metal salt of - object is washed with water, and it is attained by the washing process which is eluted selectively and acquires the 4 alkali-metal salt of (L, L)-object.

[0045] the amount of the water in a washing process -- the stereoisomer mixture of an imino disuccinic acid 4 alkali-metal salt -- receiving -- 1 - 100 weight twice -- desirable -- 2 - 20 weight twice -- that of the ** **** is good and uses for washing on filters, such as a Buchner funnel, and also it uses in a reaction vessel, and after carrying out the partial dissolution as suspension, you may once filter. The maturing temperature and maturing time in a washing process are carried out on the same conditions as a **** crystallization process.

[0046] It is an effective means, also in order for carrying out partial clearance of the (D, L)-object to prevent slurring and coloring of imino disuccinic acid and its 4 alkali-metal salt water solution and to raise the shelf life of a water solution at a precedence crystallization clearance process or a washing process.

[0047] moreover -- the case where (**) -aspartic acid and ammonia are used in an addition reaction process -- a precedence crystallization clearance process and a washing process -- setting - (D, L) object -- it is as possible as or (L, L) to acquire selectively each of the racemic mixture which consists of a (D,

D)-object. In this case, although racemic modification endures - (D, L) object in water solubility and the chelate force, it is not suitable for utilization as a biodegradability chelating agent.

[0048] the test result using the sample selectively acquired by the approach of this invention -- (L, L)-imino disuccinic acid -- biodegradability, water solubility, and the chelate force -- it became clear that it is the chelating agent which is excellent from especially any standpoint.

[0049]

[Example] Next, although an example explains this invention to a detail, this invention is not limited to the following examples.

[0050] It is 100g (1.02 mol) of maleic anhydrides to the reactor which attached example 1 agitator, the thermometer, the tap funnel, and the distillation apparatus. Anhydrous methanol 32.6g after teaching and making it dissolve at 55 to 65 degrees C (1.02 mols) It was dropped in 30 minutes. Stirring was continued for 30 minutes, holding temperature at 65 degrees C. Thus, it is 35 % of the weight to the obtained maleic-acid monomethyl ester. (L) - Aspartic-acid disodium water solution It is 45 % of the weight continuously 389g (1.02 mol). Sodium-hydroxide water solution 90.7g (1.02 mol) It added. It is reaction mixture under reflux of 105 ** 4 After carrying out time amount stirring, it is 45 % of the weight again. Sodium-hydroxide water solution 90.7g (1.02 mol) It is bottom of reflux 0.5 moreover. Time amount stirring was continued. It is this reaction mixture Further 0.5 By time-applying and carrying out moisture distilling off, desiccation disintegration of the slurry obtained is carried out by 120 ** with a spray dry method, and it is imino disuccinic acid 4 sodium salt. 361g (105% of rough yield) It obtained as a snow-white crystal. the result of the HPLC analysis using a chiral column -- this imino disuccinic acid 4 sodium salt -- 50% of (L, L)-objects 50% of (D, L)-objects from -- it is the becoming stereoisomer mixture and the byproduction of (D, D)-object was not accepted. The detailed component analysis of this imino disuccinic acid 4 sodium salt is shown in a table 1.

[0051] The same actuation as an example 1 was performed except having changed an example 2 - 4 addition-reaction time amount into the conditions shown in a table 1. A result is shown in a table 1.

[0052] The same actuation as an example 4 was performed except having changed example 5 raw material into (D)-aspartic acid. A result is shown in a table 1.

[0053] It is 100g (1.02 mol) of maleic anhydrides to the reactor which attached example 6 agitator, the thermometer, the tap funnel, and the distillation apparatus. Anhydrous methanol 100g After teaching and flowing back for 30 minutes under stirring, the excessive-amount methanol was distilled off. Thus, 45 % of the weight after adding and diluting 200g of water to the obtained maleic-acid monomethyl ester Sodium-hydroxide water solution 90.7g (1.02 mol) Subsequently it is 35 % of the weight. (L) - Aspartic-acid disodium water solution 389g (1.02 mol) It added. After stirring reaction mixture under reflux of 105 ** for 4 hours, it is 45 % of the weight again. Sodium-hydroxide water solution 90.7g (1.02 mol) In addition, stirring was continued for bottom 0.5 hours of reflux. It is this reaction mixture Further 0.5 By time-applying and carrying out moisture distilling off, desiccation disintegration of the slurry obtained is carried out by 120 ** with a spray dry method, and it is 4 sodium salt of imino disuccinic acid. 361g (105% of rough yield) It obtained as a snow-white crystal. the result of the HPLC analysis using a chiral column -- the 4 sodium salt of this imino disuccinic acid -- 50% of (L, L)-objects 50% of (D, L)-objects from -- it is the becoming stereoisomer mixture and the byproduction of (D, D)-object was not accepted. It is a table 1 about the detailed component analysis of the 4 sodium salt of this imino disuccinic acid. It is shown.

[0054] The same actuation as an example 6 was performed except having changed an example 7 - 8 addition-reaction time amount into the conditions shown in a table 1. A result is shown in a table 1.

[0055] Example of comparison 1 maleic anhydride 49.0g (0.5 mol) Water 105g It dissolved and changed into the maleic acid. It is (L)-aspartic acid to this. 66.6 g (0.5 mol) It is 48 % of the weight, cooling a solution, after adding. The sodium-hydroxide water solution was added. It reacted by stirring this reaction mixture under reflux for 20 hours. As a result of analyzing the 4 sodium salt of the imino disuccinic acid in the obtained reaction mixture in HPLC using a chiral column, it is 41% of (L, L)-objects. The byproduction of 9% of (D, D)-objects besides 50% of (D, L)-objects was accepted. It is a table 1 about the detailed component analysis of the 4 sodium salt of this imino disuccinic acid. It is

shown.

[0056] The same actuation as the example 1 of a comparison was performed except having changed the example 2 of a comparison - 3 addition-reaction time amount into the conditions shown in a table 1. A result is shown in a table 1.

[0057] The same actuation as the example 1 of a comparison was performed except having changed example of comparison 4 raw material into (D)-aspartic acid. A result is shown in a table 1.

[0058]

[A table 1]

実施例 または 比較例		付加反 応時間 [h]	成分分析値 [mol%]			IDSAの立体異性体組成比 [%]		
			IDSA	Asp	Fa	(L, L)-	(D, D)-	(D, L)-
実 施 例	1	4.0	95.5	3.3	0.3	50	0	50
	2	2.0	90.7	8.4	0.2	50	0	50
	3	8.0	97.5	1.0	0.9	50	0	50
	4	20.0	98.7	0.3	1.2	50	0	50
	5	20.0	98.8	0.2	1.2	0	50	50
	6	2.0	96.6	3.7	0.0	50	0	50
	7	4.0	97.7	2.1	0.1	50	0	50
	8	8.0	99.0	0.4	0.5	50	0	50
比 較 例	1	20.0	85.7	7.5	2.3	36	14	50
	2	2.0	14.8	85.8	0.4	50	0	50
	3	8.0	76.5	20.6	1.4	41	9	50
	4	20.0	85.7	7.5	2.3	14	36	50

[0059] However, the abbreviated name and analysis conditions in a table 1 are as follows.

(Abbreviated name)

IDSA: Iminodisuccinate Asp : Aspartic acid Fa : Fumaric acid (analysis conditions)

Component-analysis ratio: Inertsil ODS-2 A column is used. 207 nm The stereoisomer presentation ratio of Detection IDSA: MCL GEL CRSIOW A column is used. It is detection [0060] at 254 nm. 50% of - (L, L) objects acquired by the same approach as example 9 example 1 50% of (D, L)-objects from -- stereoisomer mixture of the becoming imino disuccinic acid 4 sodium salt 337g (10 mol) 150g of water - - suspending - 50 degrees C - 1 It filtered, after carrying out time amount stirring. The transparent and colorless filtrate obtained as water-soluble fractionation is 3. After mosquito moon neglect did not have a sign of discoloration or slurring, either, and was the the best for the utilization as a builder etc. After

condensing this filtrate, when evaporation to dryness is carried out by 120 °C (L, L), they are 57% of - objects, and (D, L)-object. Stereoisomer mixture of the imino disuccinic acid 4 sodium salt which consists of 43% 286g (8.5 mols, recovery 85%) It was obtained. On the other hand, it is - object when evaporation to dryness of the residue obtained as refractory nature drawing is carried out by 120 °C (L, L). 7% (D, L)-object Stereoisomer mixture of the imino disuccinic acid 4 sodium salt which consists of 93% 51g (1.5 a mol, recovery 15%) It was obtained.

[0061] The same actuation as an example 9 was performed except having changed the mass and temperature of the water used example 10-15, as shown in a table 2. A result is shown in a table 2.

[0062]

[A table 2]

実施例	水量 [g]	温度 [°C]	水溶性分画回収率 [%] (L, L:D, L)	難溶性分画回収率 [%] (L, L:D, L)
9	150	50	85 (57:43)	15 (7:93)
10	150	40	81 (57:43)	19 (21:79)
11	150	30	76 (62:38)	24 (12:88)
12	120	30	67 (69:31)	33 (12:88)
13	120	20	55 (84:16)	45 (9:91)
14	100	20	48 (96: 4)	52 (8:92)
15	80	0	32 (97: 3)	68 (28:72)

[0063] 98% after cooling radiationally the hydrolysis reaction mixture obtained in the example 16 example 1 to 50 degrees C Sulfuric acid 225g (2.25 mols) Per minute 5.0 g It added at the dropping rate. In the meantime, whenever [reaction liquid temperature] went up at 80 degrees C. It is this reaction mixture at 20 degrees C 8 Since the crystal deposited when time amount radiational cooling was carried out, it carried out the °C exception. Crystal which will consist of the stereoisomer mixture and the sodium sulfate of imino disuccinic acid if evaporation to dryness of the wet crystal obtained as refractory nature drawing is carried out by 105 °C 508g It obtained. Crystal which will consist of the stereoisomer mixture and the sodium sulfate of imino disuccinic acid on the other hand if evaporation to dryness is carried out by 105 °C after condensing the transparent and colorless filtrate obtained as water-soluble fractionation 265g It obtained. The presentation of each partition is shown in a table 3.

[0064] It was operated like the example 16 except having changed an example 17 - 22 radiational-cooling temperature, and time amount into the conditions shown in a table 3. A result is shown in a table 3.

[0065]

[A table 3]

実施例	温度 [℃]	時間 [h]	水溶性分画回収率 [%] (L, L:D, L)	難溶性分画回収率 [%] (L, L:D, L)
1 6	20	8	76 (64:36)	24 (4:96)
1 7	20	24	70 (69:31)	30 (7:93)
1 8	10	4	68 (63:37)	32 (22:78)
1 9	10	8	63 (68:32)	37 (19:81)
2 0	5	2	46 (91: 9)	54 (15:85)
2 1	5	4	32 (97: 3)	68 (28:72)
2 2	0	2	28 (86:14)	72 (36:64)

[0066] The biodegradability trial of the - (L, L) imino disuccinic acid 4 sodium salt obtained in the example 23 example 15 was performed on the following conditions according to the OECD test guideline 301 correction MITI trial. A result is shown in a table 4.

(Test condition)

Test-sample concentration: 30 mg/l active sludge: Standard active sludge active sludge concentration: 100 mg/l test temperature: 25**1 ** duration of test: 28 days [0067] On the same conditions as five to example of comparison 8 example 23, the raw ***** trial was performed about (D, D)-, (D, L)-imino disuccinic acid 4 sodium salt, and such isomer mixture. A result is shown in a table 4.

[0068]

[A table 4]

実施例 および 比較例	試験サンプル組成比 [L, L : D, D : D, L]	生分解性試験結果 [%]		
		HPLC分析	TOC換算	BOD換算
実施例23	97 : 0 : 3	100	100	100
比較例5	0 : 97 : 3	8	8	6
比較例6	0 : 0 : 100	100	98	85
比較例7	25 : 25 : 50	79	78	72
比較例8	50 : 0 : 50	100	100	92

[0069] It is the prehension ability to the calcium ion of the - (L, L) imino disuccinic acid 4 sodium salt obtained in the example 24 example 15 J.Am.Oil.Chem.Soc., 1970, 48 volumes, and 682 It measured

according to the approach given in a page. A result is shown in a table 5.

[0070] On the same conditions as nine to example of comparison 12 example 24, the prehension ability trial was performed also about (D, L)-imino disuccinic acid 4 sodium salt and isomer mixture. A result is shown in a table 5.

[0071]

[A table 5]

実施例 および 比較例	試験サンプル組成比 [L, L:D, L]	Ca ⁺⁺ 捕捉能 [CaCO ₃ mg/g換算]	
		pH 7	pH 10
実施例24	97 : 3	131	261
比較例 9	69 : 31	115	247
比較例10	50 : 50	117	237
比較例11	21 : 79	109	221
比較例12	0 : 100	102	210

[0072] According to the approach of this invention, a high yield deer can also obtain the imino disuccinic acid and its alkali-metal salt of a high grade from a maleic anhydride by short reaction time using the maleic-acid half ester prepared easily. Moreover, this invention also has the following advantages.

- (1) The aspartic acid which can come to hand as an industrial raw material, an aspartic-acid alkali-metal salt, ammonia, and these water solutions can be used.
- (2) Since there is almost no byproduction of an impurity through an addition reaction process, a hydrolysis reaction process, an evaporation-to-dryness process, or a **** crystallization process, the operation as 1 tub reaction is possible, without isolating intermediate field.
- (3) Maleic-acid half ester is the liquid of the non-volatile which is rich in water solubility and a fluidity unlike the maleic anhydride and maleic acid which were used with the conventional technique, and is very advantageous on handling.
- (4) Since there is no fear of progress of side reaction which is not desirable, such as an amidation reaction, in an addition reaction process, various combination at a mixed sequence foreword with other raw materials is possible.
- (5) Since an addition reaction ends in a short time, industrial production with high productivity is possible.
- (6) Since racemization of the specified substance does not advance at all through a process unlike the conventional technique and the specified substance which does not include (D, D)-object of difficulty biodegradability by using (L)-aspartic acid for a raw material is obtained, it is very advantageous when using as a biodegradability chelating agent.
- (7) If (L)-aspartic acid is used for a raw material, biodegradability, the chelate force, and - (L, L) object excellent in especially water solubility are selectively acquirable with elution of - object by clearance and washing of - object by precedence crystallization (D, L) (L, L) out of the mixture of (L, L)-object of the specified substance obtained, and (D, L) - object.

*** NOTICES ***

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

EXAMPLE

[Example] Next, although an example explains this invention to a detail, this invention is not limited to the following examples.

[0050] It is 100g (1.02 mol) of maleic anhydrides to the reactor which attached example 1 agitator, the thermometer, the tap funnel, and the distillation apparatus. Anhydrous methanol 32.6g after teaching and making it dissolve at 55 to 65 degrees C (1.02 mols) It was dropped in 30 minutes. Stirring was continued for 30 minutes, holding temperature at 65 degrees C. Thus, it is 35 % of the weight to the obtained maleic-acid monomethyl ester. (L) - Aspartic-acid disodium water solution It is 45 % of the weight continuously 389g (1.02 mol). Sodium-hydroxide water solution 90.7g (1.02 mol) It added. It is reaction mixture under reflux of 105 ** 4 After carrying out time amount stirring, it is 45 % of the weight again. Sodium-hydroxide water solution 90.7g (1.02 mol) It is bottom of reflux 0.5 moreover. Time amount stirring was continued. It is this reaction mixture Further 0.5 By time-applying and carrying out moisture distilling off, desiccation disintegration of the slurry obtained is carried out by 120 ** with a spray dry method, and it is imino disuccinic acid 4 sodium salt. 361g (105% of rough yield) It obtained as a snow-white crystal. the result of the HPLC analysis using a chiral column -- this imino disuccinic acid 4 sodium salt -- 50% of (L, L)-objects 50% of (D, L)-objects from -- it is the becoming stereoisomer mixture and the byproduction of (D, D)-object was not accepted. The detailed component analysis of this imino disuccinic acid 4 sodium salt is shown in a table 1.

[0051] The same actuation as an example 1 was performed except having changed an example 2 - 4 addition-reaction time amount into the conditions shown in a table 1. A result is shown in a table 1.

[0052] The same actuation as an example 4 was performed except having changed example 5 raw material into (D)-aspartic acid. A result is shown in a table 1.

[0053] It is 100g (1.02 mol) of maleic anhydrides to the reactor which attached example 6 agitator, the thermometer, the tap funnel, and the distillation apparatus. Anhydrous methanol 100g After teaching and flowing back for 30 minutes under stirring, the excessive-amount methanol was distilled off. Thus, 45 % of the weight after adding and diluting 200g of water to the obtained maleic-acid monomethyl ester Sodium-hydroxide water solution 90.7g (1.02 mol) Subsequently it is 35 % of the weight. (L) - Aspartic-acid disodium water solution 389g (1.02 mol) It added. After stirring reaction mixture under reflux of 105 ** for 4 hours, it is 45 % of the weight again. Sodium-hydroxide water solution 90.7g (1.02 mol) In addition, stirring was continued for bottom 0.5 hours of reflux. It is this reaction mixture Further 0.5 By time-applying and carrying out moisture distilling off, desiccation disintegration of the slurry obtained is carried out by 120 ** with a spray dry method, and it is 4 sodium salt of imino disuccinic acid. 361g (105% of rough yield) It obtained as a snow-white crystal. the result of the HPLC analysis using a chiral column -- the 4 sodium salt of this imino disuccinic acid -- 50% of (L, L)-objects 50% of (D, L)-objects from -- it is the becoming stereoisomer mixture and the byproduction of (D, D)-object was not accepted. It is a table 1 about the detailed component analysis of the 4 sodium salt of this imino disuccinic acid. It is shown.

[0054] The same actuation as an example 6 was performed except having changed an example 7 - 8 addition-reaction time amount into the conditions shown in a table 1. A result is shown in a table 1.

[0055] Example of comparison 1 maleic anhydride 49.0g (0.5 mol) Water 105g It dissolved and changed into the maleic acid. It is (L)-aspartic acid to this. 66.6 g (0.5 mol) It is 48 % of the weight, cooling a solution, after adding. The sodium-hydroxide water solution was added. It reacted by stirring this reaction mixture under reflux for 20 hours. As a result of analyzing the 4 sodium salt of the imino disuccinic acid in the obtained reaction mixture in HPLC using a chiral column, it is 41% of (L, L)-objects. The byproduction of 9% of (D, D)-objects besides 50% of (D, L)-objects was accepted. It is a table 1 about the detailed component analysis of the 4 sodium salt of this imino disuccinic acid. It is shown.

[0056] The same actuation as the example 1 of a comparison was performed except having changed the example 2 of a comparison - 3 addition-reaction time amount into the conditions shown in a table 1. A result is shown in a table 1.

[0057] The same actuation as the example 1 of a comparison was performed except having changed example of comparison 4 raw material into (D)-aspartic acid. A result is shown in a table 1.

[0058]

[A table 1]

実施例 または 比較例		付加反 応時間 [h]	成分分析値 [mol%]			IDSAの立体異性体組成比 [%]		
			IDSA	Asp	Fa	(L, L)-	(D, D)-	(D, L)-
実 施 例	1	4.0	95.5	3.3	0.3	50	0	50
	2	2.0	90.7	8.4	0.2	50	0	50
	3	8.0	97.5	1.0	0.9	50	0	50
	4	20.0	98.7	0.3	1.2	50	0	50
	5	20.0	98.8	0.2	1.2	0	50	50
	6	2.0	96.6	3.7	0.0	50	0	50
	7	4.0	97.7	2.1	0.1	50	0	50
	8	8.0	99.0	0.4	0.5	50	0	50
比 較 例	1	20.0	85.7	7.5	2.3	36	14	50
	2	2.0	14.8	85.8	0.4	50	0	50
	3	8.0	76.5	20.6	1.4	41	9	50
	4	20.0	85.7	7.5	2.3	14	36	50

[0059] However, the abbreviated name and analysis conditions in a table 1 are as follows.

(Abbreviated name)

IDSA: Iminodisuccinate Asp : Aspartic acid Fa : Fumaric acid (analysis conditions)

Component-analysis ratio: Inertsil ODS-2 A column is used. 207 nm The stereoisomer presentation ratio of Detection IDSA: MCL GEL CRSIOW A column is used. It is detection [0060] at 254 nm. 50% of - (L, L) objects acquired by the same approach as example 9 example 1 50% of (D, L)-objects from -- stereoisomer mixture of the becoming imino disuccinic acid 4 sodium salt 337g (10 mol) 150g of water - - suspending -- 50 degrees C -- 1 lt filtered, after carrying out time amount stirring. The transparent and colorless filtrate obtained as water-soluble fractionation is 3. After mosquito moon neglect did not have a sign of discoloration or slurring, either, and was the the best for the utilization as a builder etc. After condensing this filtrate, when evaporation to dryness is carried out by 120 ** (L, L), they are 57% of - objects, and (D, L)-object. Stereoisomer mixture of the imino disuccinic acid 4 sodium salt which consists of 43% 286g (8.5 mols, recovery 85%) It was obtained. On the other hand, it is - object when evaporation to dryness of the residue obtained as refractory nature drawing is carried out by 120 ** (L, L). 7% (D, L)-object Stereoisomer mixture of the imino disuccinic acid 4 sodium salt which consists of 93% 51g (1.5 a mol, recovery 15%) It was obtained.

[0061] The same actuation as an example 9 was performed except having changed the mass and temperature of the water used example 10-15, as shown in a table 2. A result is shown in a table 2.

[0062]

[A table 2]

実施例	水量 [g]	温度 [°C]	水溶性分画回収率 [%] (L, L:D, L)	難溶性分画回収率 [%] (L, L:D, L)
9	150	50	85 (57:43)	15 (7:93)
1 0	150	40	81 (57:43)	19 (21:79)
1 1	150	30	76 (62:38)	24 (12:88)
1 2	120	30	67 (69:31)	33 (12:88)
1 3	120	20	55 (84:16)	45 (9:91)
1 4	100	20	48 (96: 4)	52 (8:92)
1 5	80	0	32 (97: 3)	68 (28:72)

[0063] 98% after cooling radiationally the hydrolysis reaction mixture obtained in the example 16 example 1 to 50 degrees C Sulfuric acid 225g (2.25 mols) Per minute 5.0 g It added at the dropping rate. In the meantime, whenever [reaction liquid temperature] went up at 80 degrees C. It is this reaction mixture at 20 degrees C 8 Since the crystal deposited when time amount radiationnal cooling was carried out, it carried out the ** exception. Crystal which will consist of the stereoisomer mixture and the sodium sulfate of imino disuccinic acid if evaporation to dryness of the wet crystal obtained as refractory nature drawing is carried out by 105 ** 508g It obtained. Crystal which will consist of the stereoisomer mixture and the sodium sulfate of imino disuccinic acid on the other hand if evaporation to dryness is carried out by 105 ** after condensing the transparent and colorless filtrate obtained as water-soluble fractionation 265g It obtained. The presentation of each partition is shown in a table 3.

[0064] It was operated like the example 16 except having changed an example 17 - 22 radiationnal-cooling temperature, and time amount into the conditions shown in a table 3. A result is shown in a table 3.

[0065]

[A table 3]

実施例	温度 [℃]	時間 [h]	水溶性分画回収率 [%] (L, L:D, L)	難溶性分画回収率 [%] (L, L:D, L)
1 6	20	8	76 (64:36)	24 (4:96)
1 7	20	24	70 (69:31)	30 (7:93)
1 8	10	4	68 (63:37)	32 (22:78)
1 9	10	8	63 (68:32)	37 (19:81)
2 0	5	2	46 (91: 9)	54 (15:85)
2 1	5	4	32 (97: 3)	68 (28:72)
2 2	0	2	28 (86:14)	72 (36:64)

[0066] The biodegradability trial of the - (L, L) imino disuccinic acid 4 sodium salt obtained in the example 23 example 15 was performed on the following conditions according to the OECD test guideline 301 correction MITI trial. A result is shown in a table 4.

(Test condition)

Test-sample concentration: 30 mg/l active sludge: Standard active sludge active sludge concentration: 100 mg/l test temperature: 25**1 ** duration of test: 28 days [0067] On the same conditions as five to example of comparison 8 example 23, the raw ***** trial was performed about (D, D)-, (D, L)-imino disuccinic acid 4 sodium salt, and such isomer mixture. A result is shown in a table 4.

[0068]

[A table 4]

実施例 および 比較例	試験サンプル組成比 [L, L : D, D : D, L]	生分解性試験結果 [%]		
		HPLC分析	TOC換算	BOD換算
実施例23	97 : 0 : 3	100	100	100
比較例 5	0 : 97 : 3	8	8	6
比較例 6	0 : 0 : 100	100	98	85
比較例 7	25 : 25 : 50	79	78	72
比較例 8	50 : 0 : 50	100	100	92

[0069] It is the prehension ability to the calcium ion of the - (L, L) imino disuccinic acid 4 sodium salt obtained in the example 24 example 15 J.Am.Oil.Chem.Soc., 1970, 48 volumes, and 682 It measured according to the approach given in a page. A result is shown in a table 5.

[0070] On the same conditions as nine to example of comparison 12 example 24, the prehension ability trial was performed also about (D, L)-imino disuccinic acid 4 sodium salt and isomer mixture. A result is shown in a table 5.

[0071]

[A table 5]

実施例 および 比較例	試験サンプル組成比 [L, L:D, L]	Ca ⁺⁺ 捕捉能 [CaCO ₃ mg/g換算]	
		p H 7	p H 1 0
実施例24	97 : 3	131	261
比較例 9	69 : 31	115	247
比較例10	50 : 50	117	237
比較例11	21 : 79	109	221
比較例12	0 : 100	102	210

[0072] According to the approach of this invention, a high yield deer can also obtain the imino disuccinic acid and its alkali-metal salt of a high grade from a maleic anhydride by short reaction time using the maleic-acid half ester prepared easily. Moreover, this invention also has the following advantages.

- (1) The aspartic acid which can come to hand as an industrial raw material, an aspartic-acid alkali-metal salt, ammonia, and these water solutions can be used.
- (2) Since there is almost no byproduction of an impurity through an addition reaction process, a hydrolysis reaction process, an evaporation-to-dryness process, or a **** crystallization process, the operation as 1 tub reaction is possible, without isolating intermediate field.
- (3) Maleic-acid half ester is the liquid of the non-volatile which is rich in water solubility and a fluidity unlike the maleic anhydride and maleic acid which were used with the conventional technique, and is very advantageous on handling.
- (4) Since there is no fear of progress of side reaction which is not desirable, such as an amidation reaction, in an addition reaction process, various combination at a mixed sequence foreword with other raw materials is possible.
- (5) Since an addition reaction ends in a short time, industrial production with high productivity is possible.
- (6) Since racemization of the specified substance does not advance at all through a process unlike the conventional technique and the specified substance which does not include (D, D)-object of difficulty biodegradability by using (L)-aspartic acid for a raw material is obtained, it is very advantageous when using as a biodegradability chelating agent.
- (7) If (L)-aspartic acid is used for a raw material, biodegradability, the chelate force, and - (L, L) object excellent in especially water solubility are selectively acquirable with elution of - object by clearance and washing of - object by precedence crystallization (D, L) (L, L) out of the mixture of (L, L)-object of the specified substance obtained, and (D, L) - object.